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HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
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MEMORANDUM

SUBJECT: **Pirimiphos-methyl**. (List B Case No. 2535/Chemical ID No. 108102). OPPTS 860.1480: Storage Stability in Stored Grain and Processed Fractions. MRID Nos. 45311801; 45311802; 45311803; and 45311804. DP Barcode No. D272627.

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In response to data requirements discussed in the residue chemistry chapter of the HED RED (DP Barcode No. D240744; C. Swartz, 6/1/98), registrant Agriliance, LLC, has submitted storage stability data in stored grain and grain processed fractions. HED has reviewed the data to assess the potential impact of the data on reassessed tolerances and the human health risk assessment for pirimiphos-methyl.

Recommendation

The submitted storage stability data are adequate, and satisfy the requirements for OPPTS GLN

860.1480 for pirimiphos-methyl. Although residue declines were noted in several commodities, these did not affect the reassessed tolerances or the human health risk assessment for pirimiphos-methyl.

Conclusions

1. The submitted storage stability studies are adequate, and satisfy reregistration data requirements for OPPTS GLN 860.1480 for pirimiphos-methyl.

2. Residues of pirimiphos-methyl (PM) and its metabolite des-ethyl pirimiphos-methyl (DPM) are relatively stable for at least 30 months in frozen stored corn grain. The data support the results of corn grain residue trials, in which corn grain was stored frozen for at most 32 months.

3a. The data demonstrate stability of PM and DPM residues for up to 18 months of frozen storage in corn flour, corn grits and corn oil (wet and dry milled). Residues in corn meal gradually declined by approximately 20% after 18 months of storage. In corn starch, PM residues declined by approximately 20% after 16 months of storage, but there were no further declines after 40 months of storage; DPM residues in corn starch were not analyzed after 15 months of storage, due to a lack of reference material. During 15 months of storage, DPM residues were relatively stable in frozen corn starch.

3b. The submitted storage stability data support the previously reviewed corn processing study, in which samples were stored frozen for up to 18 months prior to analysis. The decline in residues in frozen corn meal and starch does not change the previous HED conclusion that no tolerances are needed in corn processing commodities other than oil.

4a. Residues of PM and DPM in stored frozen sorghum grain declined by 40 to 50% after 4 months of frozen storage. Residue recoveries from stored samples were somewhat variable between 6 and 24 months of storage, although no further declines were noted. After 55 months of frozen storage, PM residues showed a decline of approximately 10%, which is inconsistent with the analyses conducted at 1 through 24 months of storage (DPM residues were not analyzed after 24 months).

4b. Sorghum grain residue samples used to reassess the tolerance for residues in sorghum were stored frozen for up to 18 months, and therefore residue declines may have occurred.

4c. HED does not advocate an increase in the reassessed tolerance for pirimiphos-methyl residues in sorghum grain; the reassessed tolerance was based on multiple applications to grain, but BEAD usage data indicate a single application is typical [labels permit a single application, but more than one application could occur as grain is transferred].

5. Pirimiphos-methyl residues are stable in frozen sorghum flour for up to 44 months, and DPM residues are generally stable in frozen sorghum flour for up to 18 months. These data support the results of the sorghum processing study, in which flour was stored frozen for 16 months prior to analysis.
6. The submitted data do not affect the dietary component of the human health risk assessment for pirimiphos-methyl.

DETAILED CONSIDERATIONS

Background

Products containing the active ingredient pirimiphos-methyl are registered for use on stored grain and as a bulk seed treatment. In conjunction with reregistration, a number of residue chemistry studies were required to support continued use on stored grain, including additional storage stability data [OPPTS GLN No. 860.1480].

Residue data supporting the uses on stored grain, including processing studies and storage stability data, were reviewed under DP Barcode Nos. D227552; D228760; D229663; D230598; and D231449 (C. Swartz, 10/8/97). Samples from the residue trials on corn and sorghum grain were stored for up to 32 months.

In the storage stability studies, pirimiphos-methyl and metabolite R36341 (des-ethyl pirimiphos-methyl) in corn grain showed an average initial decline of 16% and 45%, respectively, in residue levels after 1 month of frozen storage; subsequent analyses at 2 and 4 months showed no further decline in residues. A similar situation was observed for residues of pirimiphos-methyl in stored sorghum grain; residues declined by 30% after 1 month of frozen storage and were stable thereafter. A steady decline in residue levels was observed only for residues of R36341 in sorghum grain; residues declined steadily from 93% at time-zero to 51% by 4 months.

In another study, storage stability data reflected grain and corn oil storage intervals of 1483-1560 days (4-4.3 years). Corn, sorghum, and wheat grain were treated in 1988 with pirimiphos-methyl at 1x the registered rate. Grain and corn oil were originally analyzed in 1988-1989 after unspecified harvest-to-sampling intervals and placed in frozen storage until a re-analysis was performed in 1993. Pirimiphos-methyl residues declined by 45% in corn grain and were relatively stable in sorghum grain and refined corn oil. There was an apparent 47% increase in pirimiphos-methyl residues in wheat grain. However, the submission did not report the storage intervals or conditions of the samples prior to the first analysis.

Storage stability data submitted with the residue data in stored grain were deemed inadequate; storage intervals did not match those incurred in residue trials, and residue declines were observed. HED was unable to determine an appropriate residue decline factor, and new studies were requested.

Present Considerations

In order to address the deficiencies noted in the 10/8/97 memorandum, the registrant Agriliance, LLC has submitted storage stability data in corn, corn processing fractions, sorghum and sorghum processing fractions [MRID Nos. 45311801; 45311802; 45311803; and 45311804]. All four studies were conducted for Agriliance by Compliance Services International in Tacoma, WA.

These studies are briefly reviewed herein, to determine their potential impact on the risk assessment or tolerance reassessment processes for pirimiphos-methyl.

Fortification/Sample Storage

Fortification of grain and processing fraction samples, followed by storage under frozen conditions, was adequately described in each of the four storage stability studies:

Corn Grain [MRID No. 45311801]

Duplicate samples of corn grain were fortified with 10 ppm pirimiphos-methyl (PM), and an additional duplicate set was fortified with 1 ppm des-ethyl pirimiphos-methyl (DPM); these samples were then placed into frozen storage at -15 (± 5) C. Samples were analyzed for PM and DPM at day 0, and after 1, 2, 4, 6, 9, 12, 18, 24 and 30 months of storage. At each interval, sample analysis sets consisted of one control sample, two samples freshly fortified with 10 ppm PM and 1 ppm DPM, and the two sets of duplicate fortified storage stability samples.

Corn Processing Fractions [MRID No. 45311802]

Duplicate samples of corn flour, grits, meal, starch, and refined oil from both wet and dry milling were fortified with 3 ppm PM, and an additional duplicate set was fortified with 3 ppm DPM; these samples were then placed into frozen storage at -15 (± 5) C. Samples were analyzed for PM and DPM at day 0, and after 1, 3, 6, 9, 12, 15 and 18 months of frozen storage, with the exception of starch, for which the final interval was 40 months, and not 18 months. Another problem with starch involved the 1 and 3 month samples, for which inconsistent results were obtained, apparently due to preparation of the fortified samples. Therefore, the study was re-initiated with new samples; the original 0-day and 1-month analyses were retained. For the new starch samples, 0-day analyses were conducted, the 1-month interval was skipped, and starch samples were analyzed at 3 months and the remaining intervals.

At each interval, sample analysis sets for each matrix consisted of one control sample, two samples freshly fortified with 3 ppm PM and 3 ppm DPM, and the two sets of duplicate fortified storage stability samples.

Sorghum Grain [MRID No. 45311803]

Duplicate samples of sorghum grain were fortified with 10 ppm PM, and an additional duplicate set was fortified with 1 ppm DPM; these samples were then placed into frozen storage at -15 (± 5) C. Samples were analyzed for PM and DPM at day 0, and after 1, 2, 4, 6, 9, 12, 18, 24 and 55 months of storage; however, residues of DPM were not quantified at 55 months, due to a lack of reference standard. At each interval, sample analysis sets consisted of one control sample, two samples freshly fortified with 10 ppm PM and 1 ppm DPM, and the two sets of duplicate fortified storage stability samples.

Sorghum Processing Fractions [MRID No. 45311804]

Duplicate samples of sorghum flour were fortified with 3 ppm PM, and an additional duplicate set was fortified with 3 ppm DPM; these samples were then placed into frozen storage at -15 (± 5) C. Samples were analyzed for PM and DPM at day 0, and after 1, 3, 6, 9, 12, 15, 18, and 44 months of storage; however, residues of DPM were not quantified at 44 months, due to a lack of reference standard. At each interval, sample analysis sets consisted of one control sample, two samples freshly fortified with 3 ppm PM and 3 ppm DPM, and the two sets of duplicate fortified storage stability samples.

Analytical Method

Pirimiphos-methyl and des-ethyl pirimiphos-methyl residues in corn, sorghum and grain processed fractions were determined using Method No. CSI-011, version -8, which codifies all previous versions of the method (i.e., versions -04 and -06), "Analytical Method for the Determination of Pirimiphos-methyl and Des-ethyl Pirimiphos-methyl in Wheat, Corn, Sorghum, and Grain Fractions by Gas Chromatography," developed by Compliance Services International. The methods are modifications of Method I in PAM, Vol. II.

Briefly, residues in grain, aspirated grain fractions and non-oily processed fractions are extracted with toluene and partitioned against water. The toluene extract is dried over sodium sulfate and concentrated by evaporating under vacuum. Residues are analyzed using gas chromatography with flame photometric detection in the phosphorus mode (GC/FPD). The stated limits of quantitation (LOQs) for the method are shown in Table 1.

Table 1. Analytical Limits of Quantitation (LOQs) for Stored Grain and Processed Fractions.

Matrix	LOQ - Pirimiphos-methyl (ppm)	LOQ - Des-ethyl pirimiphos-methyl (ppm)
Corn, grain	0.05	0.05
Flour	0.25	0.05
Grits	0.05	0.05
Meal	0.05	0.05
Starch	0.05	0.05
Oil (refined, dry milling)	0.25	0.05
Oil (refined, wet milling)	0.25	0.25
Sorghum, grain	0.05	0.05
Flour	0.05	0.05

Adequate sample calculations and representative chromatograms were included in each of the four study reports.

Method Validation Data

Untreated grain and processed fractions were used to conduct method validation studies. In addition, when storage stability samples were analyzed, freshly fortified samples were used to generate concurrent method recoveries. In method validation studies, samples were fortified with a combination of PM and DPM as shown in Table 2; samples were fortified with DPM at lower concentrations than PM, since DPM is a minor component of pirimiphos-methyl residues in stored grain. The results of the method validation data support the LOQs as stated in the study report.

[Concurrent recoveries were adequate, and are shown in detail with the results from sample analyses.]

Table 2. Method Validation Data for Grain and Processed Fractions.

Matrix	Fortification Levels, ppm (PM/DPM)	PM Recovery, ppm (% Recovery)	DPM Recovery, ppm (% Recovery)
Corn, grain	0.05 / 0.05	0.04, 0.04, 0.05 (84, 83, 105)	0.04, 0.04. 0.05 (89, 78, 97)
	5.0 / 0.2	5.0, 5.1, 5.1 (99, 102, 101)	0.19, 0.2, 0.2 (97, 93, 98)
	25 / 0.2	29, 29, 29 (115, 117, 116)	0.24, 0.19, 0.22 (95, 112, 98)
Flour	0.25 / 0.05	0.23, 0.27, 0.25 (93, 109, 100)	0.06, 0.05, 0.05 (113, 106, 104)
	5.0 / 0.2	5.2, 5.6, 5.5 (104, 112, 110)	0.22, 0.21, 0.20 (110, 104, 104)
	25.0 / 0.5	26, 27, 27 (104, 108, 108)	0.52, 0.48, 0.43 (104, 96, 85)
Grits	0.05 / 0.05	0.05, 0.05, 0.05 (98, 98, 91)	0.05, 0.05, 0.04 (96, 90, 74)
	5.0 / 0.2	4.7, 4.7, 4.9 (94, 94, 97)	0.20, 0.22, 0.20 (102, 109, 102)
	25.0 / 0.5	24, 24, 23 (96, 95, 91)	0.49, 0.48, 0.48 (98, 96, 96)
Meal	0.05 / 0.05	0.05, 0.05, 0.04 (107, 104, 85)	0.05, 0.04, 0.04 (103, 81, 75)
	5.0 / 0.2	4.8, 4.6, 4.7 (95, 92, 93)	0.20, 0.19, 0.21 (101, 96, 104)
	25.0 / 0.5	25, 23, 25 (100, 94, 101)	0.46, 0.43, 0.46 (92, 86, 91)
Starch	0.05 / 0.05	0.05, 0.04, 0.04 (98, 88, 88)	0.05, 0.04, 0.04 (86, 76, 97)
	5.0 / 0.2	4.4, 4.7, 4.5 (89, 93, 90)	0.19, 0.18, 0.19 (97, 88, 94)
	25.0 / 0.5	23, 24, 25 (91, 98, 99)	0.52, 0.45, 0.47 (103, 90, 93)
Oil (refined, dry milled)	0.25 / 0.05	0.24, 0.23, 0.23 (93, 95, 84)	0.05, 0.04, 0.05 (92, 88, 95)
	5.0 / 0.2	4.0, 4.2, 4.0 (84, 81, 81)	0.20, 0.19, 0.20 (99, 97, 100)
	25.0 / 0.5	23, 23, 23 (92, 90, 92)	0.46, 0.46, 0.46 (93, 92, 92)
Oil (refined, wet milled)	0.25 / 0.25	0.26, 0.26, 0.27 (105, 103, 108)	0.25, 0.23, 0.24 (102, 94, 95)
	5.0 / 0.5	4.5, 4.6, 4.6 (89, 93, 92)	0.50, 0.51, 0.49 (99, 102, 99)

Table 2. Method Validation Data for Grain and Processed Fractions.

Matrix	Fortification Levels, ppm (PM/DPM)	PM Recovery, ppm (% Recovery)	DPM Recovery, ppm (% Recovery)
	25.0 / 1.0	22, 23, 23 (89, 92, 91)	0.96, 0.96, 0.96 (96, 96, 96)
Sorghum, grain	0.05 / 0.05	0.06, 0.05 (115, 107)	0.05, 0.04 (103, 83)
	20.0 / 0.2	19, 20 (97, 102)	0.20, 0.19 (99, 95)
	0.05 / 0.05	0.06, 0.06 (114, 114)	0.05, 0.05 (90, 99)
Flour	0.05 / 0.05	0.05, 0.05, 0.05 (98, 96, 104)	0.04, 0.04, 0.04 (83, 75, 85)
	5.0 / 0.2	5.0, 5.0, 5.0 (101, 100, 98)	0.20, 0.22, 0.19 (101, 109, 94)
	25.0 / 0.5	24, 24, 23 (96, 94, 91)	0.48, 0.46, 0.51 (96, 91, 102)

Results of Storage Stability Analyses

Detailed results of the sorghum and corn grain and processing fractions storage stability studies are shown in Tables 3, 4 and 5.

Corn Grain

The submitted data indicate that PM and DPM residues are relatively stable for up to 30 months in stored corn grain. Although there was an initial residue decline of approximately 10% for PM and 20% for DPM after one month of storage, residues were then stable for the remainder of the study. These data support the submitted corn residue trials in which corn grain was stored up to 32 months prior to analysis; no changes are needed in the proposed reassessed tolerance of 20 ppm for residues in corn grain.

Corn Processing Fractions

The storage stability study indicates PM and DPM residues are generally stable for up to 18 months of frozen storage in corn flour, corn grits and corn oil (wet and dry milled). Residues in corn meal gradually declined by approximately 20% after 18 months of storage. In corn starch (from wet milling), PM residues declined by approximately 20% after 16 months of storage, and were not analyzed again until 40 months of storage, when residues were shown to be stable. Residues of DPM were not analyzed after 15 months of storage, due to a lack of reference material. During 15 months of storage, DPM residues appeared to be relatively stable in starch, despite re-initiation of the study and an apparent increase in residues after 3 months of storage.

The submitted storage stability data support the previously reviewed corn processing study, in which samples were stored frozen for up to 18 months prior to analysis; therefore, there is no need to change previous HED recommendations regarding reassessed tolerances for PM residues in corn grain processing fractions.

Sorghum Grain

The submitted data indicate that PM and DPM residues in sorghum grain declined by 40 to 50% after 4 months of frozen storage. Residue recoveries from stored samples were somewhat variable between 6 and 24 months of storage, although no further declines were noted. The final analysis at 55 months included only recoveries of PM, since no additional reference material was available for DPM. After 55 months of frozen storage, PM residues showed a decline of approximately 10%, which is inconsistent with the analyses conducted at 1 through 24 months of storage.

In order to determine if the data can be used to support the submitted magnitude of the residue data for sorghum grain, HED examined MRID No. 44073901 to determine the length of storage incurred for the samples which were used to set the tolerance (i.e., highest residue trial value, and HAFT, or highest average field trial). The highest residues were generated in the trial conducted in ND, following the 4th application of pirimiphos-methyl at the 1x application rate; residue samples were stored frozen for approximately 17 months. The three residue values in grain sampled immediately after the 4th application were 17.7, 16.4, and 17.1 ppm (residues of DPM were not detected, but were assumed to present at ½ the LOD, or 0.025 ppm).

Although the data suggest that residue declines may have occurred in stored sorghum grain, HED does not advocate an increase in the proposed re-assessed tolerance for pirimiphos-methyl residues in grain. The proposed tolerance of 20 ppm is based on the potential for multiple applications to stored grain [labels permit a single application, but more than one application could occur as grain is transferred]. BEAD data suggest that multiple applications are not typical, and HED considers tolerance-exceeding residues in sorghum to be unlikely, given the available usage and cultural practices data.

Sorghum Processing Fractions - Flour

The submitted data indicate PM residues are stable in frozen sorghum flour for up to 44 months, and DPM residues are generally stable in frozen sorghum flour for up to 18 months (DPM analyses were not continued beyond 18 months). In a sorghum processing study submitted in support of reregistration, sorghum processed fractions were stored frozen for 16 months prior to analysis. The results of the current storage stability study indicate that PM and DPM (a minor component of the pirimiphos-methyl residue in grain sorghum) residues did not decline in sorghum processing samples during storage incurred prior to analysis. Therefore, the subject storage stability study is adequate to support the sorghum processing study. No changes are needed for the proposed reassessed tolerances.

Conclusions

The storage stability data are adequate, and satisfy the requirements for OPPTS GLN 860.1480 for pirimiphos-methyl. Although residue declines were noted in sorghum grain, corn meal and corn starch, there is no need to change the reassessed tolerances for pirimiphos-methyl residues in stored grain. In addition, the dietary (food only) component of the dietary exposure assessment remains unchanged; pirimiphos-methyl residues in grain sorghum essentially did not make a contribution to dietary exposure, since consumption of sorghum is so low.

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7509C:CSwartz:RRB1:CM2:Rm 722H:703 305 5877:07/09/01

Table 3. Storage Stability and Concurrent Recoveries of PM and DPM from Corn Grain and Processing Fractions.

Corn Fraction	Residue	% Recoveries After Frozen Storage, [Concurrent Method Recoveries (%)]											
		0-Day	1-Month	2-Months	3-Month	4-Month	6-Month	9-Month	12-Month	15-Month	18-Month	24-Month	30-Month
Grain	PM	98.6, 89.8 [101, 96.1]	81.0, 76.9 [99.8, 97.4]	82.7, 88.5 [102, 104]		79.8, 76.3 [93.7, 96.9]	83.4, 83.1 [91.2, 93.1]	81.0, 82.3 [95.1, 96.3]	82.4, 85.6 [89.0, 93.8]		86.2, 80.8 [104, 99.6]	83.4, 87.4 [100, 109]	83.7, 88.9 [98.6, 98.0]
	DPM	99.1, 117 [96.8, 83.1]	76.2, 74.5 [100, 102]	77.5, 92.2 [92.3, 113]		73.3, 77.3 [104, 94.5]	91.3, 80.2 [108, 106]	83.6, 64.3 [75.9, 98.7]	91.8, 80.3 [108, 102]		91.5, 87.7 [117, 104]	95.0, 77.3 [104, 87.7]	81.3, 76.7 [93.3, 106]
Flour	PM	110, 114 [108, 97.8]	107, 90.4 [108, 111]		91.9, 91.8 [96.7, 96.7]		88.8, 87.9 [91.1, 93.0]	91.8, 98.0 [95.2, 103]	91.1, 90.4 [97.0, 93.5]	108, 101 [112, 110]	109, 114 [108, 114]		
	DPM	94.4, 83.9 [79.8, 85.1]	106, 98.3 [87.9, 95.2]		95.1, 90.6 [93.6, 93.5]		114, 104 [90.1, 96.5]	101, 110 [96.3, 109]	85.9, 90.0 [93.9, 90.5]	102, 104 [111, 110]	106, 109 [116, 117]		
Grits	PM	102, 98.9 [109, 106]	86.4, 83.9 [104, 104]		75.3, 67.6 [90.8, 95.6]		87.0, 97.6 [100, 104]	97.7, 81.6 [106, 114]	78.5, 85.8 [99.3, 103.2]	99.5, 99.8 [91.1, 103.1]	92.8, 91.6 [103, 104]		
	DPM	81.6, 92.1 [99.4, 87.8]	81.6, 94.8 [99.2, 105]		63.6, 74.5 [92.9, 92.6]		104, 92.6 [95.7, 104]	87.2, 91.9 [109, 105]	78.6, 74.6 [97.0, 102]	90.5, 88.1 [91.2, 105]	95.4, 97.7 [108, 105]		
Meal	PM	102, 98.4 [99.1, 98.1]	95.2, 95.2 [87.7, 98.7]		92.0, 83.7 [91.7, 91.8]		83.1, 88.3 [94.3, 90.1]	85.8, 82.2 [97.6, 103]	92.8, 92.4 [99.7, 104]	101, 90.2 [112, 104]	89.1, 85.7 [104, 102]		
	DPM	103, 93.3 [94.9, 90.8]	99.8, 85.9 [94.6, 99.9]		88.9, 86.2 [96.7, 92.9]		114, 117 [104, 104]	96.8, 100.1 [114, 109]	96.0, 94.8 [102, 106]	92.4, 96.7 [111, 104]	91.1, 89.5 [112, 111]		40 Months
Starch	PM	97.0, 93.9 [88.3, 98.8]	96.4, 90.7 [93.6, 94.4]		102, 93.4 [105, 98.4]		97.4, 100 [107, 107]	87.3, 74.8 [94.4, 102]	107, 108 [116, 114]	85.4, 77.1 [105, 98.6]			95.3, 82.4 [93.1, 92.4]
	DPM	92.0, 91.6 [88.5, 100]	88.5, 98.1 [99.187.9]		113, 110 [104, 89.1]		104, 95.3 [99.0, 94.7]	102, 96.5 [95.9, 102]	91.7, 115 [106, 106]	100.2, 96.2 [104, 104]			
Oil (Dry)	PM	116, 73.9 [103, 82.6]	95.9, 104 [89.6, 102]		85.8, 90.8 [95.0, 94.4]		92.9, 93.8 [97.4, 98.4]	96.0, 92.7 [96.1, 99.6]	89.2, 91.8 [92.0, 87.4]	102, 102 [100, 105]	88.5, 86.8 [87.8, 88.8]		
	DPM	91.2, 108 [96.2, 93.5]	109, 93.1 [92.9, 94.2]		92.5, 86.6 [94.3, 102]		116, 111 [102, 99.1]	102, 106 [95.1, 104]	106, 98.5 [95.9, 92.8]	108, 110 [101, 108]	101, 91.8 [93.8, 93.5]		
Oil (Wet)	PM	99.1, 97.0 [92.0, 93.5]	94.6, 105 [94.9, 97.8]		85.6, 88.7 [94.6, 94.7]		97.2, 93.1 [90.0, 92.5]	94.8, 94.0 [92.2, 102]	91.7, 94.4 [95.3, 92.2]	94.4, 91.4 [92.0, 90.6]	90.5, 90.3 [92.2, 91.9]		
	DPM	108, 94.4 [114, 102]	84.6, 93.0 [102, 93.8]		85.9, 87.0 [93.9, 99.0]		115, 115 [91.4, 89.5]	91.1, 101 [97.2, 106]	95.0, 98.6 [99.0, 101]	94.5, 99.4 [93.8, 97.5]	93.8, 96.2 [96.4, 95.2]		

Table 4. Storage Stability and Concurrent Recoveries of PM and DPM from Sorghum Grain.

Residue	% Recoveries After Frozen Storage, [Concurrent Method Recoveries (%)]									
	0-Day	1-Month	2-Month	4-Month	6-Month	9-Month	12-Month	18-Month	24-Month	55-Month
PM	89.3, 94.7 [96.5, 99.3]	65.0, 64.2 [98.7, 102]	56.8, 66.1 [104, 105]	54.5, 55.6 [91.8, 95.8]	72.4, 68.0 [98.4, 101]	64.4, 58.1 [108, 112]	62.0, 59.7 [95.3, 96.7]	76.3, 74.6 [91.8, 102]	60.5, 62.6 [101, 97.0]	91.7, 94.5 [110, 112]
DPM	115, 81.2 [102, 109]	83.5, 70.5 [110, 92.9]	44.2, 67.3 [99.9, 90.8	42.4, 58.1 [96.4, 100]	57.8, 64.7 [103, 100]	71.5, 75.5 [99.1, 105]	61.0, 51.3 [108, 102]	64.2, 77.5 [101, 112]	52.5, 60.8 [113, 88]	

Table 5. Storage Stability and Concurrent Recoveries of PM and DPM from Sorghum Flour.

Residue	% Recoveries After Frozen Storage, [Concurrent Method Recoveries (%)]								
	0-Day	1 Month	3-Month	6-Month	9-Month	12-Month	15-Month	18-Month	44-Month
PM	99.6, 101 [99.8, 111]	100, 111 [104, 109]	93.5, 97.5 [93.7, 96.0]	94.1, 97.5 [97.4, 88.9]	101, 106 [106, 104]	96.7, 98.1 [103, 104]	107, 101 [110, 105]	109, 106 [99.9, 98.8]	99.6, 91.7 [109, 108]
DPM	93.6, 118 [108, 103]	109, 98.0 [103, 106]	86.4, 88.9 [87.9, 93.5]	108, 115 [88.2, 87.0]	113, 118 [113, 112]	102, 101 [101, 102]	105, 97.9 [103, 100]	96.3, 112.3 [104, 102]	